PATENT SPECIFICATION

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PROVISIONAL SPECIFICATION

Improvements in or relating to the Heat Treatment of Magnesium Base Alloys

We, MAGNESIUM ELEKTRON LIMITED, a British Company, of Lumm's Lane, Clifton Junction, near Manchester, do hereby declare the nature of this invention to be as follows:—

This invention relates to the heat treatment of magnesium base alloys containing 4—6% zinc and between 0.5% and 1% of zirconium in a condition which (together with the magnesium which contains it) is readily soluble in an aqueous solution of hydrochloric acid consisting of 30 ccs. of HCl (specific gravity 1.16) to 85 ccs. of water, sufficient acid being added during dissolution to maintain the initial concentration.

It has been found that the mechanical properties of such alloys can be improved by heat treatment at temperatures which result in the reprecipitation of the zirconium and/or zinc in a finely divided condition. We have found that particularly good results can be obtained by carrying out this heat treatment at temperatures of about 180—200° C. This temperature range may be extended up to about 300° C. If it is desired to effect a stress relief annealing of castings made from these alloys, the heat treatment must be carried out at a temperature of at least 300—330° C. However, it is to be expected and has been found that a heat treatment at temperatures of 300—330 until most of the zirconium is precipitated would result in the precipitation of the zirconium in a coarse form accompanied by a reduction in mechanical properties.

We have now made the surprising discovery that a heat treatment of such alloys at temperatures of about 300—350° C. for comparatively short periods followed by a heat treatment at a considerably lower temperature avoids the precipitation of the zirconium in a coarse condition and at the same time produces appreciably higher mechanical properties than are obtainable by a heat treatment at the lower temperature alone. This short-term heat treatment at 300—350° C. also effects adequate stress relief of the casting.

According to the present invention, therefore, the alloys are subjected to a heat treatment at a temperature above 300° C. for a period short enough to avoid appreciable precipitation of zirconium in a coarse condition and the alloy is subsequently subjected to a further heat treatment at a temperature at which the zirconium and/or zinc are precipitated in a finely divided condition. The treatment at the higher temperature does not result in any appreciable solution of any zinc-rich beta phase which may be present.

The heat treatment at the higher temperature is preferably carried out for a period of about 1—6 hours the lower temperature generally requiring a longer duration of treatment. The treatment of the alloys for 2 hours at 330° C. has given particularly good results while achieving the complete stress relief commonly required. The treatment at the low temperature may be effected for a period of 12—48 hours at temperatures of 250—150° C., the lower temperature again generally involving the longer duration of treatment. A treatment at 180° C. for 24 hours has given particularly good results.

The alloys of the present invention may contain between 0.05—0.3% of rare earth metals in the form of mischmetal or its individual components or mixtures thereof.

The alloys of the present invention must, in accordance with the invention

described in the Specification of the Applicant Company's British Patent No. 511,137, be substantially free from elements which form insoluble high melting point compounds with zirconium such as aluminium, manganese, silicon, tin, cobalt, nickel and antimony. Other elements which do not form such insoluble compounds may, however, be included, e.g. cadmium in proportions up to 5%, mercury up to about 1%, calcium up to 0.2% and thorium and silver up to 0.5% each and in total. Also quite small quantities or traces of copper, bismuth, barium and lead may be present.

Dated this 6th day of January, 1948.

For the Applicants,
S. MATTHEWS,
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COMPLETE SPECIFICATION

Improvements in or relating to the Heat Treatment of Magnesium Base Alloys

We, MAGNESIUM ELEKTRON LIMITED, a British Company, of Lumn's Lane, Clifton Junction, near Manchester, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the heat treatment of magnesium base alloys of the kind containing at least 90% magnesium, 4—6% zinc, and between 0.5% and 1% of zirconium in a condition which (together with the magnesium which contains it) is readily soluble in an aqueous solution of hydrochloric acid consisting of 30 ccs. of HCl (specific gravity 1.16) to 85 ccs of water, sufficient acid being added during dissolution to maintain the initial concentration, with or without cadmium up to 5% and/or quite small quantities or traces of one or more of the following viz., rare earth metals, silver, thorium, mercury, beryllium, copper bismuth, barium, lead and calcium. The maximum quantity of rare earth metals, silver, thorium, copper, bismuth, and barium, will be 0.5% each and in total; of mercury and lead 1%; and of beryllium maximum solubility; and calcium 0.2%. The alloys of the present invention must, in accordance with the invention described in the Specification of the Applicant Company's British Patent No. 511,137, be substantially free from elements which precipitate zirconium from a magnesium melt, such as aluminium, manganese, silicon, tin, cobalt, nickel and antimony.

It has been found that the mechanical properties of alloys of the kind above referred to can be improved by heat treatment at temperatures which result in the precipitation of the zirconium and/or zinc in a finely divided condition. We have found that particularly good results can be obtained by carrying out this heat treatment at temperatures of about 180—200° C. If it is desired to effect a stress relief annealing of castings made from these alloys, the heat treatment may often be carried out at a temperature of about 300—330° C. However, it is to be expected and has been found that a heat treatment at temperatures of 300—330° C. would result in a reduction in mechanical properties presumably due to the precipitation of the zirconium in a coarse form.

We have now made the surprising discovery that a heat treatment of such alloys at temperatures of about 300—350° C. for comparatively short periods followed by a heat treatment at a considerably lower temperature produces appreciably higher mechanical properties than are obtainable by a heat treatment at the lower temperature alone, probably by avoiding the precipitation of the zirconium in a coarse condition. This short-term heat treatment at 300—350° C. also effects adequate stress relief of the casting.

The coarse condition of zirconium precipitation is easily observable in a photomicrograph when the heat treatment is sufficiently prolonged but is not so easily distinguishable in the early stages of precipitation. Nevertheless even these early stages of precipitation will result in loss of mechanical properties in the fully heat treated casting. Similarly the finely divided precipitation can also be more definitely measured by the final mechanical properties. Therefore for the purpose of the present invention the periods and temperatures of the two parts of the heat treatment are selected in order to avoid loss of proof stress, or more accurately to ensure obtaining at least the following minimum figures viz., 10 tons per square inch 0.1% proof stress for an alloy containing 4% zinc and proportionately up to 11.0 tons per square inch for an alloy containing 6% zinc. A few quite simple tests are therefore sufficient to in-

dicate the required treatment according to the present invention for any particular alloy. Thus if an alloy be subjected to the two treatments and the final proof stress is below that above quoted, it can be assumed that either (a) the first part of the treatment is too long or too short and/or (b) the second part of the treatment is too short. Therefore a further test is made with a shortened or lengthened period for the first part of the treatment and/or a more extended treatment for the second part of the treatment, being guided by the fact that in the first part of the treatment a period more than 3 hours at 400° C. is generally too long, and more than six hours at 330° C. is generally too long, and more than about 12 hours at 260° C. is similarly undesirable. The period of the first part of the treatment may therefore be from 3 hours at 400° C. proportionately to 12 hours at 260° C. In the second part of the treatment a period of at least 6 hours is required at 240° C. and at least 48 hours at 150° C. and at least 72 hours at 100° C. No loss of proof stress occurs by extending the period of the second part of the treatment.

The proof stress figures above quoted are for sand cast test bars made in accordance with Directorate of Technical Development Specification No. 59B, the proof stress being determined from a curve drawn by plotting extensometer readings against a continuously increasing loading applied to the test bar.

It is to be understood that in applying the invention to castings other than test bars the appropriate times and temperatures for the alloy can be ascertained for test bars and then the same times and temperatures applied to the castings.

Another method of selecting the appropriate periods of treatment is based on a comparison of the proof stress obtained by the double treatment according to this invention and the proof stress obtained by a treatment of the same alloy at 180° C. for 24 hours. Thus if the double treatment gives a proof stress greater than that given by the low temperature alone, the double treatment may be considered to be satisfactory. For example a double treatment of 2 hours at 330° C. and 24 hours at 180° C. may give 10.5 tons per square inch proof stress as compared with an average of about 9.9 tons per square inch proof stress after only 224 hours at 180° C. If a double treatment gives a less proof stress than the low temperature treatment alone. then the periods and temperatures must be readjusted on the lines indicated above i.e. avoiding too long or too short periods for the first part of the treatment, or too short periods for the second part, or similarly varying the temperatures within the quoted ranges. It will be clear that shortening the period of the first part of the treatment is equivalent to reducing the temperature of that part of the treatment, and in either part of the treatment lengthening the period is equivalent to increasing the temperature.

According to the present invention, therefore, the alloys are subjected to a heat treatment at a temperature between 260° C. and 400° C. (generally above 300° C.) and the alloy is subsequently subjected to a further heat treatment at a temperature between 100° C. and 240° C., the temperature and period of both these treatments being selected so as to produce one or both of the following results:—

1. When applied to a test bar according to Directorate of Technical Development Specification No. 59B, this test bar will have a 0.1% proof stress of at least 10 tons per square inch at 4% zinc, this figure rising proportionally with increasing zinc content to 11.0 tons per square inch at 6% zinc.

2. A 0.1% proof stress greater than the 0.1% proof stress produced by subjecting the alloy to a temperature of 180° C. for 24 hours.

The treatment at the higher temperature does not result in any appreciable solution of any zinc-rich beta phase which may be present.

The heat treatment at the higher temperature is preferably carried out for a period of about 1—12 hours dependent upon the temperature as above indicated, the lower temperature generally requiring a longer duration of treatment. The treatment of the alloys for 2 hours at 330° C. has given particularly good results while achieving the complete stress relief commonly required. The treatment at the low temperature may be effected for a period of 6—48 hours at temperatures of 240—150° C.. or even down to 100° C., the lower temperature again generally involving the longer duration of treatment. A treatment at 180° C. for 24 hours has given particularly good results. The treatment at the lower temperatures may however be continued for a number of days.

The advantage achieved by the present invention is illustrated by way of example in the following experimental results obtained by heat treatment of an alloy of the following composition:—

Zirconium (soluble in hydrochloric acid as hereinbefore described) - - -	0.7%
Zinc - - - - - - -	4.5%
Magnesium - - - - -	Remainder

Time and Temperature of Heat Treatment.	0.1% Proof Stress.	Ultimate Tensile Stress.	Elongation % on 2″.
	(tons/sq.in.)		
As Cast - - - - - -	7.9	16.0	9.5
24 hrs. at 180° C. - -	9.9	17.7	9.0
24 hrs. at 330° C. - -	8.6	16.4	9.5
2 hrs. at 330° C. - -	9.3	17.7	13.5
2 hrs. at 330° C. + 24 hrs. at 180° C. - - -	10.5	18.5	10.0
24 hours at 330° C. + 24 hrs. at 180° C. - -	9.7	15.5	4.0

The castings may be cooled between the first and second heat treatments and after the second heat treatment by air cooling, water quenching or oil quenching.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A method for the heat treatment of magnesium base alloys of the kind defined which consists in subjecting the alloys to treatment at a temperature between 260—400° C., and subsequently subjecting the alloy to a further heat treatment at a temperature between 100° C. and 240° C., the temperature and period of both treatments being so selected as to produce a 0.1% proof stress greater than the 0.1% proof stress produced by subjecting the alloy to a temperature of 180° C. for 24 hours.

2. A method for the heat treatment of magnesium base alloys of the kind defined which consists in subjecting the alloys to treatment at a temperature between 260—400° C., and subsequently subjecting the alloy to a further heat treatment at a temperature between 100° C. and 240° C., the temperature and period of both treatments being so selected that when applied to a test bar according to Directorate of Technical Development Specification No. 59B, this test bar will have a 0.1% proof stress of at least 10 tons per square inch at 4% zinc, this figure rising proportionally with increasing zinc content to 11.0 tons per square inch at 6% zinc.

3. A method as claimed in claim 1 or 2 wherein the period of the first part of the treatment is from 3 hours at 400° C. proportionally to 12 hours at 260° C.

4. A method as claimed in claim 1, 2, or 3 wherein the period of the second part of the treatment is from at least 6 hours at 240° C. to at least 48 hours at 150° C. and at least 72 hours at 100° C.

5. A method as claimed in claim 1 substantially as hereinbefore described.

6. A magnesium base alloy of the kind defined when subjected to the heat treatment claimed in claim 1 or 2.

Dated this 16th day of December, 1948.

For the Applicants,
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